

# FCS02 – SOP for General Laboratory Procedures for FCU

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## 1. Scope

- 1.1. This document details what is expected and required of personnel for sampling, analysis, documentation, and reporting of suspected controlled dangerous substances. Also, this document includes how to ensure the quality of the whole process including: evidence control, analytical procedures, instrument/equipment performance, use of balances, chemicals and reagents, casework documentation, report writing, proficiency and competency testing, analytical method validation and verification, laboratory audits, deficiency of analysis, and health and safety.

## 2. Background

- 2.1. To establish the best practices for operations within the Forensic Chemistry Unit and to ensure conformance to the requirements of the Department of Forensic Sciences (DFS), the accreditation standards under ISO/IEC 17025:2017, and any supplemental standards.

## 3. Safety

- 3.1. Read Material Safety Data Sheets to determine the safety hazards for chemicals and reagents used in the standard operating procedures.

- 3.2. Wear personal protective equipment (e.g., lab coat, gloves, mask, eye protection), when carrying out standard operating procedures.

## **4. Materials Required**

- 4.1. As required to perform analyses.

## **5. Standards and Controls**

- 5.1. As required to perform analyses.

## **6. Calibration**

- 6.1. As required to perform analyses.

## **7. Procedures**

- 7.1. Methods of Analysis - Drug Identification

7.1.1. Herein are the minimum standards for the forensic identification of commonly seized drugs. It is recognized that the correct identification of a drug or chemical depends on the use of an analytical scheme based on validated methods and the competence of the analyst. The FCU requires the use of multiple uncorrelated techniques.

- 7.1.2. Categorizing analytical techniques

7.1.2.1. Techniques for the analysis of drug samples are classified into three categories (see Table 1), based on their maximum potential discriminating power. However, the classification of a technique may be lower, if the sample, analyte, or mode of operation diminishes its discriminating power.

- 7.1.3. Examples of diminished discriminating power may include:

7.1.3.1. An infrared spectroscopy technique applied to a mixture which produces a combined spectrum, or

7.1.3.2. A mass spectrometry technique which only produces molecular weight information.

Category A	Category B	Category C
Infrared Spectroscopy	Capillary Electrophoresis	Color Tests
Mass Spectrometry	Gas Chromatography	Fluorescence Spectroscopy
Nuclear Magnetic Resonance Spectroscopy	Ion Mobility Spectrometry	Immunoassay
Raman Spectroscopy	Liquid Chromatography	Melting Point
X-ray Diffractometry	Microcrystalline Tests	Ultraviolet Spectroscopy
	Pharmaceutical Identifiers	
	Thin Layer Chromatography	

**Table 1.** SWGDRUG Categories of Analytical Techniques

#### 7.1.4. Identification Criteria

- 7.1.4.1. When a validated Category A technique is incorporated into an analytical scheme, at least one other technique (from either Category A, B or C) shall be used.
- 7.1.4.2. When a Category A technique is not used, at least three different validated techniques shall be employed. Two of the three techniques shall be based on uncorrelated techniques from Category B.
- 7.1.4.3. For the use of any method to be considered of value, test results shall be considered “positive” (*i.e.*, it must meet the acceptance criteria defined in the method validation and operating protocol). When possible, data from a test result should be compared to data generated from a reference material which has been analyzed under the same analytical conditions. While “negative” test results provide useful information for ruling out the presence of a particular drug or drug class, these results have no value toward establishing the forensic identification of a drug.

- 7.1.4.3.1. When “negative” or “inconclusive” test results are achieved, an additional test of similar or

higher discriminating category may be used in order to identify the presence of a substance. Additionally, you may consult with a senior chemist for further guidance.

7.1.4.4. The laboratory shall employ quality assurance measures to ensure the results correspond to the exhibit. Example measures are:

7.1.4.4.1. The use of two separate samplings,

7.1.4.4.2. Sample identification procedures, such as bar-coding and witness checks,

7.1.4.4.3. Good laboratory practices (e.g., positive and negative controls, one sample opened at a time, procedural blanks).

7.1.4.5. In cases where tandem techniques are used, e.g., gas chromatography-mass spectrometry, liquid chromatography-diode array ultraviolet spectroscopy, they will be considered as separate techniques provided that the results from each are used.

7.1.4.6. The chosen analytical scheme shall demonstrate the identity of the specific drug present and shall minimize false positive and false negative identifications. Where a scheme has limitations, this shall be reflected in the final interpretation.

## 7.2. Methods of Analysis – Analogue and Structure Class Determination

7.2.1. Classification as a controlled substance analogue involves the evaluation of the similarity of structure of a chemical compound to a known controlled substance.

7.2.1.1. Structural determinations are evaluated on:

7.2.1.1.1. The interpretation of mass spectra for an unknown versus known drug compound, or

7.2.1.1.2. The interpretation of mass spectra for an unknown versus literature-reported chemical structure if no current standard exists.

7.2.1.1.3. Documentation shall be kept on the evaluation of similarities between chemical compounds, including a discussion of how the compounds are similar and how they are different. Evaluation of similarity is a subjective matter and opinions may differ. A consultation among experts may be necessary.

7.2.1.1.4. Structural comparisons in a forensic laboratory may be limited to the structural class and functional group, ring or chain substitutions. As examples, isomers, homologues, salt forms, atomic substitutions, esters, and ethers may be considered. The scope of comparison conducted should be made clear in the report.

### 7.3. Reporting

7.3.1. All conclusions and opinions expressed in written or oral form shall be based on sufficient supporting evidence, data, or information, as defined by laboratory procedures.

7.3.2. The basis of any conclusion should be completely documented in the case notes and summarized in the written report and subject to the laboratory's review policy.

7.3.3. Conclusions and opinions reported shall be accurate and clear enough so that other laboratory testing personnel can understand and replicate them.

7.3.4. The report should clearly indicate what elements of the legal requirements were evaluated and what elements were not evaluated.

7.3.5. The scope of opinions and conclusions reported, in either written or oral form, shall not go beyond the knowledge, training and experience of the analyst.

#### 7.3.6. Case Reporting Process

##### 7.3.6.1. Metropolitan Police Department (MPD) Case Send-Out

7.3.6.1.1. The FCU report for MPD-submitted cases will be sent to the MPD general reporting email (cid-evidence.reports@dc.gov) and a copy will be placed in the USAO shared drive.

7.3.6.2. Other Agency Case Send-Out

7.3.6.2.1. Other agencies must establish points of contact for the agency and must specify who will receive reports.

7.3.6.2.2. FCU Reports will be sent out to the agency points of contact.

7.3.6.2.3. Supplemental case information may be provided upon agency request and approval from the FCU Unit Manager.

7.3.7. The FCU Report packet, titled Report of Examination / Supplemental Report of Examination will consist of:

7.3.7.1. If the method performed is accredited under the scope of the laboratory, then the accreditation stamp for ILAC-MRA / ANAB will be placed at the bottom of the report page.

7.3.7.2. If a method performed is NOT accredited under the scope of the laboratory, then the accreditation stamp for ILAC-MRA / ANAB will NOT be placed at the bottom of the report page. Additionally, a comment within the Notes section will explicitly state that the method used is not accredited and for information purposes only.

7.3.8. Supplemental Reports:

7.3.8.1. When additional evidence is received for analysis after the original report has been released, a supplemental laboratory report will be issued and will be marked with the word "Supplemental" and read in the title "Supplemental". Additionally, a note will be added to the report to include a reference to the original and/or previously released report(s).

7.3.9. Amended Reports:

7.3.9.1. Once a Report of Examination / Supplemental Report of Examination has been issued, the laboratory will represent any required material amendments in the form of an Amended Report of Examination / Amended Supplemental Report of Examination.

- 7.3.9.2. The word “Amended” is added to the title of the report / notification.
- 7.3.9.3. The reason why the report is amended will be described within the notes block of the Amended Report of Examination / Amended Supplemental Report of Examination.
- 7.3.9.4. The date on all pages of the Amended Report of Examination / Amended Supplemental Report of Examination will reflect the date of the amended report / notification.
- 7.3.9.5. The amended report / notification will be reviewed as per *FCS06 – SOP for Reviewing Reports*.
- 7.3.10. A Review packet shall consist of the following items, as appropriate:
  - 7.3.10.1. The FCU report
  - 7.3.10.2. The original request for analysis
  - 7.3.10.3. The chemist’s drug worksheet
  - 7.3.10.4. The case’s activity communication log, unless it is accessible during the Discovery process from the Laboratory Information Management System (LIMS)
  - 7.3.10.5. PDF versions of case communication emails, or equivalent
  - 7.3.10.6. Instrumental data from the case, typically in this order:
    - 7.3.10.6.1. Physical identification data
    - 7.3.10.6.2. Fourier Transform Infra-Red (FT-IR) spectroscopy data
    - 7.3.10.6.3. Gas Chromatography Mass Spectrometry (GC-MS) data
    - 7.3.10.6.4. Gas Chromatography Flame Ionization Detection (GC-FID) data
    - 7.3.10.6.5. Miscellaneous Info, such as:
      - 7.3.10.6.5.1. DEA-7 forms
      - 7.3.10.6.5.2. Pictures from the case

7.3.10.6.5.3. Supporting documentation

7.3.10.6.6. FCU Review Checklist

7.4. Evidence Control

7.4.1. Receiving and identifying evidence

7.4.1.1. The FCU shall maintain records of requests for analysis and of the respective items of evidence. A unique identifier shall be assigned to each case submitted for testing and the information pertaining to it will be retained in its own electronic case file. For chain-of-custody purposes, the evidence shall be compared to the submission documentation, any significant observations of irregularity shall be documented in the case file or record, and the submitter informed promptly. This file or record shall include at minimum the following:

7.4.1.1.1. Submission documents or copies (or electronic equivalent), such as the DEA-7 form,

7.4.1.1.2. Identity of party requesting analysis and the date of request,

7.4.1.1.3. Description of items of evidence submitted for analysis,

7.4.1.1.4. Identity of the person who physically delivered the evidence, along with date of submission,

7.4.1.1.5. Chain of custody record (or electronic equivalent), and

7.4.1.1.6. Unique case identifier.

7.4.2. Integrity of Evidence

7.4.2.1. Evidence shall be properly secured (e.g., sealed). Appropriate storage conditions shall ensure that, insofar as possible, the composition of the seized material is not altered. All items shall be safeguarded against loss or contamination. Any alteration of the evidence (e.g., repackaging) shall be documented. Procedures shall be implemented to assure that samples are and remain properly labeled throughout the



analytical process (see *DOM10 – Evidence Handling Procedures*).

7.4.2.2. Evidence shall only be actively worked when at least two personnel are present. This does not apply to secondary evidence (e.g., chemical washes or extracts), that are running on instruments during analysis.

7.4.2.3. When performing active investigations on a piece of evidence and the analyst must leave the room temporarily, the evidence must first be secured in a personal locker or other secure location.

#### 7.4.3. Storage of Evidence

7.4.3.1. Access to the evidence storage area shall be granted only to persons with authorization and access shall be controlled. A system shall be established to document a chain of custody for evidence in the laboratory (see *DOM10 – Evidence Handling Procedures*).

#### 7.4.4. Disposition of Evidence

7.4.4.1. Records shall be kept regarding the disposition (e.g., return, destruction, conversion to another use) of all items of evidence.

7.4.4.2. This may be accomplished through the chain of custody under normal circumstances.

#### 7.4.5. Documentation Retention Procedures

7.4.5.1. All laboratory records such as analytical results, measurements, notes, calibrations, chromatograms, spectra and reports shall be retained in a secure fashion.

#### 7.4.6. Evidence Accessioning Procedure

7.4.6.1. The DFS Central Evidence Unit (CEU) is responsible for the initial entering of evidence into the LIMS system, where practicable.

7.4.6.2. The FCU Lead Chemist or designee will receive evidence from CEU and transfer it to the Drug Vault, where it will be assigned to an analyst.

- 7.4.6.3. There shall always be at least two people who ensure transfer of evidence from CEU or other agency to the FCU evidence vault. A record of either an iris scan or sign in shall be maintained.

## 7.5. Analytical Procedures

### 7.5.1. Analytical procedures for drug analysis

- 7.5.1.1. The FCU shall have and follow documented analytical procedures.
- 7.5.1.2. The FCU shall have in place protocols for the sampling of evidence.
- 7.5.1.3. Work practices shall be established to prevent contamination of evidence during analysis.
- 7.5.1.4. The FCU shall have and follow documented guidelines for the acceptance and interpretation of data.
- 7.5.1.5. The FCU shall monitor the analytical processes using appropriate blanks, controls, or reference materials.
- 7.5.1.6. Reference materials and reference data are critical to demonstrating the validity of quantitative and qualitative test results. A positive test result shall meet the acceptance criteria defined in the method validation and operating protocol. In descending order of preference, the acceptance criteria should be based on:
  - 7.5.1.6.1. Comparison to data obtained from a suitable drug reference material analyzed under the same analytical conditions as the test/case sample.
  - 7.5.1.6.2. The reference material may be analyzed contemporaneously with test/case sample.
  - 7.5.1.6.3. As part of routine quality control, e.g., daily check solutions.
  - 7.5.1.6.4. At a previous date (e.g., method validation, in-house library).

7.5.1.6.5. Comparisons to external reference data may be used where a reference material is unavailable. External reference data shall be shown to be fit for purpose. The veracity of the data shall be considered and assessed. Factors to consider include:

7.5.1.6.5.1. Origin of the data

7.5.1.6.5.2. Validation of the data

7.5.1.6.5.3. Peer review of the data

7.5.1.6.5.4. Comparability of analytical conditions.

7.5.1.6.6. The use of external reference data rather than a reference material should be documented and where applicable the limitation expressed within the report.

7.5.1.6.7. When neither reference materials nor external reference data are available, structural elucidation techniques may be employed providing the analyst has the appropriate skills for their interpretation. Such interpretations shall be made only by analysts competent in structural elucidation interpretation. The absence of a reference material and external data shall be documented and the impact on the interpretation of reported results assessed.

7.5.1.7. Analytical procedures shall be validated.

7.5.1.8. When analysts determine the identity of a drug in a sample, they shall employ quality assurance measures to ensure the results correspond to the exhibit.

## 7.5.2. Assessment of reference materials

7.5.2.1. Reference materials shall, where possible, be traceable to SI units of measurement, or to certified reference materials (CRM). For seized drugs, this requirement is difficult to fulfill because the concept of traceability for drug standards is not

internationally established and CRM's for drug analysis are not readily available or affordable.

7.5.2.2. Note: A certificate does not necessarily define a material as a CRM.

7.5.2.3. The FCU must ensure that each reference material is fit for purpose prior to use as a reference material.

7.5.2.4. To be fit for purpose, the reference material must meet the following minimum specifications:

7.5.2.4.1. The material shall be purchased from an ISO Guide 34 certified entity, if practicable.

7.5.2.4.2. Each new material shall be analyzed as per standard drug analysis to indicate:

7.5.2.4.2.1. Approximate gas chromatographic retention time, as appropriate

7.5.2.4.2.2. Mass spectral or other pertinent analytical results

7.5.2.4.2.3. Comparison of defining feature data to reference collection, published literature, or vendor-sourced information of standards

7.5.2.4.3. This assessment shall be done on each lot of reference material.

7.5.2.4.4. This assessment shall be completed prior to or alongside casework analysis as appropriate.

7.5.2.4.5. Fit for purpose for qualitative work requires an assessment of chemical identity (structure, identifiable mass peaks, etc.).

7.5.2.4.6. Fit for purpose for quantitative work requires an assessment of purity and its associated uncertainty of measurement.

- 7.5.2.4.7. These parameters may be described in a certificate, statement of analysis, data sheet or label supplied with the material or may be determined by in-house analysis or reference to published literature.
- 7.5.2.4.8. The laboratory shall assess the reliability of the information supplied with a reference material even if the material meets the definition of a CRM.
  - 7.5.2.4.8.1. For reference materials obtained from a provider accredited under ISO Guide 34, the information contained in the accompanying certificate is considered reliable and can be accepted as correct if the material is stored in accordance with the manufacturer's instructions. In these circumstances the assessment need not include analysis.
  - 7.5.2.4.8.2. For reference materials obtained from a provider not accredited under ISO Guide 34 the identity and purity information supplied by the provider shall be verified by analysis. Other information may be evaluated as needed.
- 7.5.2.4.9. Examples of verification of chemical identity by analysis:
  - 7.5.2.4.9.1. Analysis and comparison of the results to peer-reviewed published data, data produced by a laboratory accredited under ISO/IEC 17025:2017, or to data

produced from a previously verified reference material.

7.5.2.4.9.2. Evaluation of data from in-house structural elucidation analysis of the material.

7.5.2.4.10. Examples of verification of purity by analysis utilizing validated methods:

7.5.2.4.10.1. Quantitative Nuclear Magnetic Resonance (NMR) Spectroscopy

7.5.2.4.10.2. Quantitative UltraViolet (UV)-Visible Spectroscopy

7.5.2.4.10.3. Comparison to previously verified material

7.5.2.4.11. When verification by analysis is not possible, this shall be documented and where applicable the limitation expressed within the report.

7.5.2.4.12. Where a reference material has no or limited supporting documentation or is produced in-house (by synthesis or from a case sample), then the chemical identity shall be determined in sufficient detail to demonstrate that it is fit for purpose. In addition, for quantitative work, the purity and associated uncertainty of measurement shall also be determined.

7.5.2.5. Reference materials should have an expiration date.

7.5.2.5.1. If the material is not supplied with an expiration date, one should be assigned at the first assessment. If the expiration date passes before the material is fully used, then the material can be re-assessed, and the expiry date extended. The laboratory protocol for extending expiration dates shall be documented and should include analysis of the material.

7.5.2.5.2. The procedure for extending expiration dates includes the following steps:

7.5.2.5.2.1. A sample of the material with the expiration date to be extended shall be analyzed using GC-MS and/or GC-FID and compared to a non-expired material.

7.5.2.5.2.2. The expired material and non-expired material must meet the GC-MS analysis criteria in section 7.8.1.4. of FCS01 – *SOP for Detecting Controlled Dangerous Substances*.

7.5.2.5.2.3. The new expiration date for the reassessed standard shall be set at 3 months from the date of reassessment.

7.5.2.5.2.4. The results of this reassessment shall be released in a memo which shall be placed in the FCU Shared Drive along with the relevant data used for the reassessment.

7.5.2.5.2.5. Standards may be reassessed multiple times to further extend the expiration date and a standard does not need to be expired before it undergoes a reassessment.

7.5.2.5.2.6. If expiry dates are not assigned to reference materials, the laboratory must have a documented protocol for assessing the validity of the

reference material each time it is used.

7.5.2.6. Reference materials shall only be used for the purpose defined by the laboratory. For example, a reference material may be deemed suitable for qualitative but not quantitative determinations.

7.5.2.7. For quantitative determinations, different batches of reference material should be used for calibration and quality control. Where this is not practicable, the material can be sub-divided and each part assigned a specific purpose.

7.5.2.8. The assessment and purpose of a reference material shall be documented. The documentation shall include the name of the individual who performed the assessment, the date of assessment, verification test data, and details of all reference materials and reference data used.

### 7.5.3. Color Test Reagents

7.5.3.1. Each color test will be assigned a unique lot number when it is created (or when opened, if it is manufacturer prepared).

7.5.3.2. The lot number shall take the following format:

7.5.3.2.1. Initialism of the color test used, e.g.,

7.5.3.2.1.1. CT for Cobalt Thiocyanate

7.5.3.2.1.2. MQ for Marquis Reagent

7.5.3.2.1.3. DL for Duquenois-Levine Reagent

7.5.3.2.1.4. MY for Mayer's Reagent

7.5.3.2.1.5. FC for Ferric Chloride

7.5.3.2.1.6. ER for Ehrlich's Reagent

7.5.3.2.1.7. SN for Sodium Nitroprusside

7.5.3.2.2. Date of preparation (e.g., YYYYMMDD format)



7.5.3.2.3. Preparer's initials (or MP if manufacturer prepared)

7.5.3.2.4. Example of a lot number for Brandon Paul Jones preparing a Marquis Reagent on April 2, 2018, would be MQ20180402BPJ.

7.5.3.3. Reagents used for color tests will be tested prior to use by someone other than the preparer.

7.5.3.4. Each lot of color test reagents will be retested quarterly or more often, if necessary.

7.5.3.5. Expiration dates for color test reagents will be set as 3 months from the last quarterly test (or from the preparation date if no additional quarterly retests have been performed).

## 7.6. Instrument/Equipment Performance

### 7.6.1. Instrument Performance

7.6.1.1. Instruments shall be routinely monitored to ensure that proper performance is maintained.

7.6.1.2. Monitoring shall include the use of reference materials, test mixtures, calibration standards, blanks, etc.

7.6.1.3. Instrument performance monitoring shall be documented.

7.6.1.4. The manufacturer's operation manual and other relevant documentation for instrumentation should be readily available.

### 7.6.2. Equipment

7.6.2.1. Only suitable and properly operating equipment shall be employed.

7.6.2.2. Equipment performance parameters should be routinely monitored and documented.

7.6.2.3. An annual Preventative Maintenance shall be performed to ensure instrument reliability and conformance to manufacturer's standards.

- 7.6.2.4. The manufacturer's operation manual and other relevant documentation for each piece of equipment should be readily available.
- 7.6.2.5. If instruments are moved from the normal operating positions, their performance shall be verified prior to entering service again.
- 7.6.2.6. If an instrument needs to be removed from the laboratory, *DOM13 – Health and Safety* shall be followed, in specific the Equipment Release Certification will be attached to the instrument after decontamination.

## 7.7. Use of Balances

### 7.7.1. General operational guidelines:

- 7.7.1.1. Don appropriate PPE (gloves).
- 7.7.1.2. Visually inspect balance to ensure clean; clean if necessary, with a wipe or brush and let dry prior to use.
- 7.7.1.3. Press internal calibration button, if applicable.
- 7.7.1.4. Tare the balance.
- 7.7.1.5. Record test weights (for each week the balance is in use).
  - 7.7.1.5.1. These values are used to assess the annual Uncertainty in Measurement
  - 7.7.1.5.2. Uncertainty in Measurement is assessed using Process Uncertainty, *i.e.*, the balance plus human error, recorded over the year to include temperature and user variations.
  - 7.7.1.5.3. Test weights must be no more than the below mentioned specifications:

Balance Class	Weights	Acceptable Range
Analytical	200g	199.9-200.1g
	10g	9.95-10.05g
	2g	1.95-2.05g
	0.1g	0.09-0.11g
	0.01g	0.009-0.011g
Top Loading	10,000g	9,995-10,005
	2,000g	1999-2001g
	200g	199-201g
	10g	9.9-10.1g
	2g	1.9-2.1g

7.7.1.5.4. If the test weights are outside the acceptance specifications of a test mass, the analyst shall follow Section 5.8 Malfunctioning Equipment of *DOM05 – Procedures for Instrument Checks and Maintenance*

7.7.1.5.5. The balance shall be put back into service upon subsequent demonstration of measuring weights within acceptance range

## 7.7.2. Weighing Evidence

7.7.2.1. Record the weight of the primary container, as applicable. This is the gross weight.

7.7.2.2. Remove the exhibit from the primary container as much as practicable. Record the weight of the empty primary container. This is the package weight. The net weight is the package weight subtracted from the gross weight.

7.7.2.3. Calculations performed for weights shall be rechecked by the analyst to ensure accurate data transcription.

7.7.2.4. Remove sample(s) from the exhibit for testing. Either weigh this sample individually or weigh the original exhibit, whichever is deemed more appropriate by the analyst.

### 7.7.3. Reporting Weights

7.7.3.1. Record weight values as displayed on the balance.

7.7.3.2. In each report, ensure the uncertainty of the balance used is listed (or alternatively, the lowest uncertainty for the balance class in the lab).

### 7.7.4. Reporting Volumes

7.7.4.1. Volumes of liquids may be reported during the process of casework as an approximate value and will be treated in general as a description. Note – the weight of the total evidence item will be taken and quantified as under 7.7.2.

7.7.4.2. In situations where an accurate volume is necessary, *e.g.*, as per customer request, an uncertainty of the measurement device and serial number of the device will be recorded in the case notes.

7.7.4.3. Class A glassware will be used for quantitative analyses as per *FCS15 – SOP for Quantitation of Heroin using GC-FID* and serial numbers and/or identifiers will be recorded in the case notes. All glassware will be valid for 10 years upon opening the package and inspected for noticeable defects before use as per *FCS17 – Procedure for Cleaning Glassware*. Recalibration of Class A glassware is not necessary.

## 7.8. Chemicals and Reagents

### 7.8.1. General Guidelines

7.8.1.1. Chemicals and reagents used in drug testing shall be of appropriate grade for the tests performed.

7.8.1.2. The efficacy of all test reagents shall be checked prior to their use in casework. Results of these tests shall be documented.

7.8.1.3. Chemical and reagent containers should be dated and initialed when received and when first opened.

7.8.1.4. Chemical and reagent containers shall be labeled as to their contents.

### 7.8.2. Laboratory Prepared Reagents

- 7.8.2.1. The method of preparation for all laboratory-prepared chemical reagents shall be recorded in a laboratory notebook, or electronic equivalent.
- 7.8.2.2. Any chemical reagent produced within the laboratory shall follow a documented formulation.
- 7.8.2.3. Documentation for reagents prepared within the laboratory shall include identity, concentration (when appropriate), date of preparation, identity of the individual preparing the reagents, storage conditions (if appropriate) and the expiration date.
- 7.8.2.4. Each reagent will also be marked with a lot number with the following format: YYYYMMDDInitials of preparer (Example: 20171204BPJ).
- 7.8.2.5. A Reagent Control Logbook will be maintained to document reagent preparation, testing and retesting.
- 7.8.2.6. If any reagents fail testing, all reagents from the same lot will be disposed of immediately.
- 7.8.2.7. Chemists will ensure that all their reagents are not expired and have been tested in the last quarterly test before using the reagents in casework.

### 7.9. Casework Documentation

- 7.9.1. Documentation shall contain sufficient information to allow a peer to evaluate case notes and interpret the data.
- 7.9.2. Evidence handling documentation shall include chain of custody, information regarding packaging of the evidence upon receipt, the initial weight/count of evidence to be examined (upon opening), a description of the evidence and communications regarding the case.
- 7.9.3. Analytical documentation should include procedures, standards, blanks, observations, test results and supporting documentation including charts, graphs, photos, and spectra generated during an analysis.
  - 7.9.3.1. Lot/Batch numbers of critical reagents used during a test shall be documented in the case notes. Routine solvents and

corresponding batch information will be retained in an accessible logbook.

- 7.9.4. Casework documentation shall be preserved in an electronic format in a controlled shared drive.

## 7.10. Report Writing

- 7.10.1. Reports issued by laboratories shall be accurate, clear, and objective. These reports shall include the following information:

7.10.1.1. Title of report (Report of Examination or Supplemental Report of Examination).

7.10.1.2. Identity and location of the testing laboratory

7.10.1.3. Unique case identifier (on each page)

7.10.1.4. Clear identification of the end of the report (e.g., Page 3 of 3)

7.10.1.5. Submitting agency

7.10.1.6. Date of receipt of evidence (also referred to as FCU Submission Date)

7.10.1.7. Analysis start date, analysis end date, and report date

7.10.1.8. Descriptive list of submitted evidence

7.10.1.9. Identity and signature (or electronic equivalent) of analyst

7.10.1.10. Results / conclusions

7.10.1.11. A list of analytical techniques employed

7.10.1.12. Sampling

7.10.1.13. Uncertainty (if applicable to result)

7.10.1.14. Each technical page shall include the initials of the Chemist who performed the test.

7.10.1.15. If elements listed above are not included on the report, the laboratory shall have documented reasons (*i.e.*, specific accreditation, customer or jurisdictional considerations), for not doing so.

7.10.1.16. **Note:** If in extraordinary cases when circumstances of the case and analytical processes used are fully documented but where methods are employed without prior performance verification, the report shall explicitly state that the test result is not obtained through the use of a validated procedure.

7.10.1.17. **Note:** Analysis start date is defined as the date the chemist opens the item of evidence and begins technical analysis. Analysis end date is defined as the date the chemist completes the technical analysis. Report date is defined as the date the chemist submits the report for technical review. If technical or administrative review requires edits, the report date will be updated to reflect the newest submission. Issue date is defined by the date the administrative review is completed and signed (see administrative checklist). Distribution date is defined as the date that the report is delivered to the customer.

#### 7.10.2. Case Review

7.10.2.1. The FCU has policies establishing protocols for technical and administrative case review (see §7.3 of *FCS06 – SOP for Reviewing Reports*).

7.10.2.2. The FCU has a documented policy for resolving case review disagreements between analysts and reviewers (see *FCS06 – SOP for Reviewing Reports*).

#### 7.11. Proficiency and Competency Testing

7.11.1. The FCU has a documented competency testing and proficiency testing program, including documented protocols for monitoring the competency and proficiency of its analysts. The FCU will utilize the procedures outlined in *FCS13 – Procedures for Proficiency Testing*.

#### 7.12. Analytical Method Validation and Verification

7.12.1. Method validation is required to demonstrate that methods are suitable for their intended purpose (see *DOM04 – Procedures for Validating Technical Procedures*

#### 7.13. Laboratory Audits

7.13.1. The FCU shall follow *DOM06 – Internal and External Audits* to ensure on-going quality of the laboratory.

## 7.14. Deficiency of Analysis

7.14.1. In the course of examining seized drug samples and related materials, the FCU may encounter some operations or results that are deficient in some manner. For these situations, the FCU shall follow *DOM07 – Practices for Quality Corrective Action* to address deficiencies or unapproved deviations from established policy or procedures in an analysis.

## 7.15. Health and Safety

7.15.1. The FCU follows *DOM13 - DFS Health and Safety Manual* and supplemental program guidelines.

# 8. Sampling

## 8.1. Introduction

8.1.1. Sampling evidence is the most important initial step in forensic drug analysis. One must be sure that what is sampled is truly representative of the total population. The analyst must take into consideration the homogeneity (or lack thereof) among drug packaging (bags, packets, capsules, etc.) and its contents. Careful visual inspections and personal experience are essential in determining the proper sampling procedure.

8.1.2. For items containing multiple specimens, statistically-based sampling models (e.g., percent based or hypergeometric distribution) will allow the analyst to analyze a portion of the specimens and subsequently make statistical inferences about the population. In these instances, an inference to the entire population will be drawn and the number of specimens that were analyzed will be indicated on the Report.

8.1.3. This document addresses minimum recommendations for sampling of seized drugs for qualitative analysis.

8.1.4. **NOTE:** For the purpose of this document the use of the term “statistical” refers to “probability-based.”

8.1.5. The principal purpose of sampling in the context of this recommendation is to answer relevant questions about a population by examination of a portion of the population (e.g., What is the net weight of the population? What portion of the units of a population can be said to contain a given drug at a given level of confidence?)



- 8.1.6. By developing a sampling strategy and implementing appropriate sampling schemes, a laboratory will minimize the total number of required analytical determinations, while assuring that all relevant legal and scientific requirements are met.

## 8.2. General Sampling

- 8.2.1. Every effort should be made to avoid handling evidence repeatedly. The material should be sampled and immediately sealed. If necessary, the evidence may be closed and maintained in short term storage until the analysis is complete.
- 8.2.2. In order to minimize detailed labeling on small items such as very small metal foil packets, plastic bags or plastic bag corners, they may be secured in a bandolier of tape, which is then labeled. If needed, items may be placed in an additional plastic bag which can be sealed, fully labeled, and properly documented in the case notes.
- 8.2.3. For chemical analyses, a representative sample shall be removed from the specimen. When sample size allows, testing should be applied on separate samplings of the material.
- 8.2.4. Where practicable, a **separate** sample of the exhibit shall be taken for each test. For example, one sample of a bag shall be used for presumptive color spot testing, one for GC-MS or GC-FID.

## 8.3. Sampling Strategy

- 8.3.1. Sampling may be statistical or non-statistical:
  - 8.3.1.1. In many cases, a non-statistical approach may suffice. The sampling plan shall provide an adequate basis for answering questions of applicable law (e.g., Is there a drug present in the population? Are statutory enhancement levels satisfied by the analysis of a specified number of units?)
  - 8.3.1.2. If an inference about the whole population is to be drawn from a sample, then the plan shall be statistically based and limits of the inference shall be documented.
  - 8.3.1.3. Statistically selected units shall be analyzed to meet the minimum recommendations for forensic drug identification if statistical inferences are to be made about the whole population (see Hypergeometric Sampling Plan and Percent-Based Sampling Plan).

#### 8.4. Multiple Specimens

- 8.4.1. If all specimens are not analyzed, the number of those that are fully analyzed will be recorded in the case notes.
- 8.4.2. Weights and autosampler vial numbers will be associated with specific specimens by the use of sub-numbering in the case notes.
- 8.4.3. Within any sampling scheme, Administrative or Hypergeometric, if the first set of observations determines that more than one population is present, further samples from each population must be taken.
- 8.4.4. When multiple balances are required to record weights within one item of casework, the sum of the samples taken and analyzed should meet the requirement of the selected sampling plan.

#### 8.5. Residue Specimens (<10mg)

- 8.5.1. Residues are samples which are either too small to be weighed accurately or that which remains. Residues can be sampled by mechanical means (e.g., shaking or scooping) or chemical means (e.g., rinsing with solvent). Case notes must reflect the method by which the sample was removed.
- 8.5.2. When possible, a sample should be removed while leaving a portion of the residue intact.
- 8.5.3. When it is not possible to redeposit and return the residue as received, the extract used in analysis will be returned to the evidence.

#### 8.6. Sampling Scheme

- 8.6.1. The sampling scheme is an overall approach which includes population determination, selection of the sampling plan and procedure and, when appropriate, sample reduction prior to analysis.
- 8.6.2. Population Determination:
  - 8.6.2.1. The population determination shall take into account all typical forms and quantities in which exhibits may appear.
  - 8.6.2.2. A population can consist of a single unit or multiple units.
  - 8.6.2.3. A multiple unit population shall be separated into items based on the units which are similar in relevant visual characteristics.

### 8.6.3. Sampling Plan:

8.6.3.1. Depending upon the inference to be drawn from the analysis for a multiple unit population, the sampling plan may be statistical or non-statistical

8.6.3.1.1. Statistical approaches are applicable when inferences are made about the whole population. For example:

8.6.3.1.1.1. The probability that a given percentage of the population contains the drug of interest or is positive for a given characteristic.

8.6.3.1.1.2. The total net weight of the population is to be extrapolated from the weight of a sample.

8.6.3.1.2. Non-statistical approaches are appropriate if no inference is to be made about the entire population.

### 8.6.3.2. Administrative Sampling Plan (non-statistical)

8.6.3.2.1. The administrative sampling plan will be used in cases to answer a specific legal question. If more specimens than listed below need to be analyzed for successful prosecution, additional analysis utilizing the hypergeometric sampling plan will be conducted.

#### 8.6.3.2.2. Sampling Plan

8.6.3.2.2.1. One specimen will be randomly selected and fully analyzed.

8.6.3.2.2.2. All remaining specimens will be left intact in case further analysis is required.

8.6.3.2.2.3. Exceptions to this plan may occur only at the discretion of the

Technical Supervisors in  
consultation with the Unit  
Manager.

#### 8.6.3.3. Hypergeometric Sampling Plan

- 8.6.3.3.1. Hypergeometric sampling is a statistically-based model involving a defined confidence level with an associated probability of finding failures in a population. The hypergeometric model is used for specimens with no significant markings or labels (e.g., the contents of plastic bags and bag corners, vials, and glassine packets). This model may be used when the item requires a quantitative analysis.
- 8.6.3.3.2. Hypergeometric sampling may be used when additional analysis is requested for successful prosecution.
- 8.6.3.3.3. The appropriate number of specimens within the population will be randomly selected to give a 95% level of confidence that at least 90% of the population contains the analyte in question.
- 8.6.3.3.4. Record the number of specimens indicated by the table below along with an indication of the statistical relevance of the number in the case notes.
- 8.6.3.3.5. Each specimen sampled will be analyzed separately and fully, unless otherwise directed by the customer.

**Table 2.** Hypergeometric Table for sampling of test items.

Population (N) N <sub>max</sub> =1000	Proportion of Positives = 90% (Confidence Level=95%)	Population (N) N <sub>max</sub> =1000	Proportion of Positives = 90% (Confidence Level=95%)
1-10	ALL	34	18
11	9	35	18
12	9	36	19
13	10	37	19
14	11	38	20
15	12	39	20
16	12	40	18
17	13	41	18
18	14	42	18
19	15	43	19
20	12	44	19
21	13	45	20
22	14	46	21
23	14	47	21
24	15	48	21
25	16	49	22
26	16	50-59	23
27	17	60-69	23
28	18	70-79	24
29	18	80-89	25
30	15	90-99	25
31	16	100-199	27
32	17	200-1000	28
33	17		

8.6.3.4. When a single unit or bulk population is to be analyzed, the issue of homogeneity shall be addressed within the sampling plan.

8.6.3.4.1. One sample is sufficient if the bulk material is homogeneous, or if it is made so by the analyst.

8.6.3.4.2. If the bulk material is not homogeneous, several samples from different locations may be necessary to ensure that the test results are representative of the bulk material and to avoid false negative results.

8.6.3.5. Percent-Based Sampling Plan

8.6.3.5.1. A percent-based approach to sampling may be employed, as directed by customer request. The specific percent to be sampled will be noted within the technical notes, e.g., worksheet, and

the total number of units tested and total number of units not tested will be noted.

8.6.3.5.2. A percent-based system may be developed as part of a request, including determination of acceptance criteria for the unit determination. If no prior customer request is made for what defines a unique unit within a population, then the chemist will make a decision based upon their experience.

8.6.3.5.3. As requested by the customer, either each item within the sampled population will be tested, or a composite will be made of the samples and a single test on that composite will be made.

8.6.3.5.4. In cases where a composite shall be made, the chemist will first test each of the selected samples (from the percent sampled population) with a screening technique prior to making a composite (Category A, B, or C).

#### 8.6.4. Sampling Procedure:

8.6.4.1. Establish the procedure for selecting the number of units that will comprise the sample:

8.6.4.1.1. For non-statistical approaches select a sample appropriate for the analytical objectives.

8.6.4.1.2. For statistical approaches, select a random sampling.

8.6.4.2. Select a random sample:

8.6.4.2.1. A random sample is one selected without bias. Computer generated random numbers or random number tables are commonly employed for such tasks and these should be included in the sampling plan.

8.6.4.2.2. Random sampling of items using random number tables may not be practical in all cases. In these instances, an alternate sampling plan shall be designed and documented to approach

random selection. A practical solution involves a “black box” method, which refers to one that will prevent the sampler from consciously selecting a specific item from the population (e.g., all units are placed in a box and the samples for testing are selected without bias).

#### 8.6.5. Sample Reduction

8.6.5.1. Sample reduction may be applied in cases where the weight or volume of the selected units is too large for laboratory analysis.

### 8.7. Analysis

#### 8.7.1. Statistically-selected samples:

8.7.1.1. Each unit comprising the sample shall be analyzed to meet minimum recommendations for forensic drug identification, if statistical inferences are to be made about the whole population.

#### 8.7.2. Non-statistically-selected samples:

8.7.2.1. Minimum recommendations for forensic drug identification shall be applied to at least one unit of the sample.

### 8.8. Documentation

8.8.1. Inferences drawn from the application of the sampling plan and subsequent analyses shall be documented.

### 8.9. Reporting

8.9.1. Herein are the minimum standards for the forensic identification of commonly seized drugs. It is recognized that the correct identification of a drug or chemical depends on the use of an analytical scheme based on validated methods and the competence of the analyst. The FCU requires the use of multiple uncorrelated techniques.

## 9. Calculations

9.1. See Table 2 for hypergeometric sampling plan calculation table.

## 10. Uncertainty of Measurement

10.1. See *FCS21 – Procedure for Uncertainty in Measurement*

## 11. Limitations

11.1. See specific method SOP for limitations on analytical processes.

11.2. Limitations must be clearly conveyed within the laboratory report.

## 12. Documentation

12.1. FCU Examination Worksheets

12.2. FCU Laboratory Report

## 13. References

13.1. This document is adapted from recommendations made by the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations Edition 7.1 (2016-06-9) for the use by the Department of Forensic Sciences (DFS) Forensic Chemistry Unit (FCU) in the District of Columbia.

13.2. OSAC Registry Standard: ASTM E2548-11e1 Standard Guide for Sampling Seized Drugs for Qualitative and Quantitative Analysis (Seized Drugs Subcommittee, April 3, 2017) – ASTM E2458-16

13.3. Forensic Chemistry Unit Quality Assurance Manual (Current Version)

13.4. DFS Departmental Operations Manuals (Current Versions)

13.5. FCU Standard Operating Procedures (Current Versions)